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Test Report T62-3-1

DETERMINATION OF NITROGEN IN NITROCELLULOSE BY
INFRARED SPECTROPHOTOMETRY

September 1961

FRANKFORD ARSENAL

U.S. ARMY ORDNANCE

PHILADELPHIA 37, PA

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DETERMINATION OF NITROGEN IN NITROCELLULOSE BY INFRARED SPECTROPHOTOMETRY

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I. INTRODUCTION

Nitrogen in nitrocellulose is usually determined by the DuPont nitrometer method (2, 24) or by titration (25, 31). The nitrometer method is accurate but considerable working time is required per sample and the cost and hazard of using the large amount of mercury is objectionable. The titration methods are less time-consuming than the nitrometer method but are not as accurate, and preparation and frequent standardization of reagents is required. One of the most satisfactory of the titration methods according to an interlaboratory study (19) is that of Pierson and Julian (31).

There is a need for improved methods for the determination of nitrogen in nitrocellulose. Therefore, an investigation was undertaken to develop an infrared method.

II. SUMMARY

Previous infrared studies of nitrocellulose and attempts to determine nitrocellulose and the nitrogen content of nitrocellulose by infrared are reviewed. An infrared method for the determination of nitrogen in nitrocellulose is proposed whereby the sample is dissolved in tetrahydrofuran by standing overnight and the transmittance of the band at 6 microns measured. Various factors affecting the method were considered. The results for the proposed method compare well with the results obtained by the nitrometer method. The precision of the method is about ± 0.09%.

III. RECOMMENDATIONS

It is recommended that the proposed method be adopted by Ordnance laboratories as an alternate procedure to the DuPont nitrometer method.

IV. STUDY

A. NATURE OF NITROCELLULOSE

To understand the problem of the infrared determination of nitrogen in nitrocellulose it is necessary to understand the nature of nitrocellulose.

Nitrocellulose is made by nitrating cellulose, a substance which consists of a large number of beta glucose anhydride units joined together by ether linkages through 1 and 4 positions. The number of repeating units in the molecule varies from several hundred to several thousand, depending on the source of the cellulose and its treatment. The formula of cellulose is best represented as follows (8):
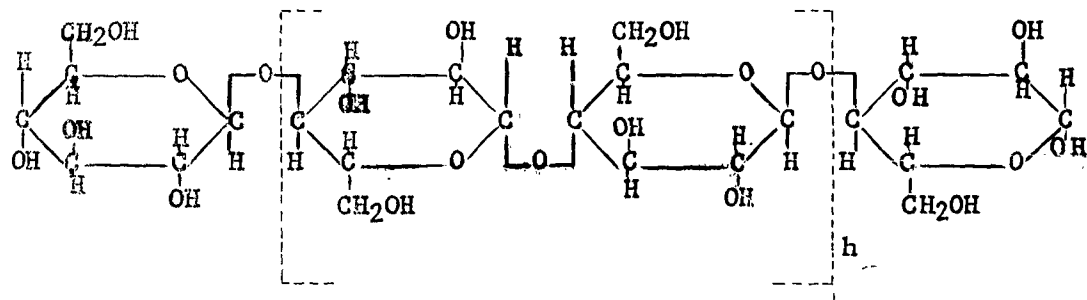
Conversion of cellulose to nitrocellulose is an esterification which results in the hydroxyl groups being replaced by ONO_2 groups (the ONO_2 groups are best represented as $-O-N\begin{smallmatrix}O\\O\end{smallmatrix}$ (29)). It can be seen from the formula of cellulose that in each glucose residue of the cellulose chain there are three hydroxyl groups (two secondary and one primary). Frequently, in the literature, mention is made of mononitrocellulose, dinitrocellulose, and trinitrocellulose, indicating that one, two or all three hydroxyl groups have been replaced (the theoretical composition of these substances is 6.75%, 11.11%, and 14.14% nitrogen respectively). Actually, since the hydroxyl groups are replaced at random, the mono- and dinitro compositions cannot be considered true compounds.

Nitrocellulose containing up to about 13.8% nitrogen is made commercially by treatment of cellulose with a mixture of nitric and sulfuric acids (16, 29). Nitrocellulose of greater than 13.8% nitrogen can ordinarily only be obtained by the use of special nitrating mixtures such as a mixture of nitric acid, phosphoric acid, and phosphorus pentoxide (29). Nitrocellulose containing less than 10.5% nitrogen is of limited commercial importance because it is difficultly soluble in organic solvents and does not have desirable physical properties (16). Nitrocellulose used in ammunition and explosives contains about 12 to 13.8% nitrogen.

B. PREVIOUS INFRARED STUDIES OF NITROCELLULOSE AND ATTEMPTS TO DETERMINE
NITROCELLULOSE AND THE NITROGEN CONTENT OF NITROCELLULOSE BY INFRARED

In order to develop an infrared method for the determination of nitrogen
in nitrocellulose, it was necessary to understand the significance of the
different bands and review previous infrared studies of nitrocellulose.

Despite the fact that nitrocellulose is such an important industrial
item, no literature survey has been made of the infrared studies on the
material. Two authoritative books on the infrared spectra of organic
compounds (5, 35) and two treatises on nitrocellulose (23, 29) mention the
subject only casually.

The first infrared study of nitrocellulose seems to have been made by
Herzog and Laski (17) who used a microradiometer as the indicating instrument
and worked with film produced by evaporating an amyl acetate solution of
nitrocellulose to dryness.

Freymann and Freymann (12) and Champetier and Clement (7) compared the
infrared spectrum of nitrocellulose with those of other nitro compounds and
noted the similiarity to nitrous esters.

Nikitin (26) immersed samples of nitrocellulose in a mixture of carbon
tetrachloride and carbon disulfide and measured the absorption between
1 and 2.5 microns. Samples nitrated between 3 and 12% nitrogen showed
an absorption band at 1.44 microns whose intensity increased with the degree
of nitration. This band was attributed to OH not disturbed by the hydrogen
bond. It was stronger when the number of nitrate groups was greater because
the bulky nitrate groups increased the distance between the cellulose chains
and thus ruptured the hydrogen bonds. Nitrocellulose containing 13.7%
nitrogen had no band at 1.44 microns because practically all the OH groups
were nitrated. The same investigator (27) noted that the sharp band at
1.44 microns in dinitrocellulose film lost 50% of its intensity when the
film was dissolved in a mixture of ethyl alcohol and ether, presumably of
exhaustive hydrogen bonding between the nitrocellulose and ethyl alcohol.
Dissolution in acetone caused complete disappearance of the 1.44 band because
of the exhaustive hydrogen bonding. Nikitin also found (28) that nitration
of cellulose caused the appearance of free OH bands at 1.91 and 2.07 microns
as well as at 1.44 microns and that hydrogen bonding was produced in dioxane
and pyridine as well as in acetone and ethyl alcohol. The 1.44 micron band
in dinitrocellulose was displaced by 100 cm^{-1} in acetone and by 580 cm^{-1} in
dioxane.

Rowen and Plyler (39) recorded the infrared spectrum of a film obtained by evaporating an ethyl acetate solution of the nitrocellulose. They noted that water in the film increased the absorption at the 6.07 micron band.

Barchewitz, Henry, and Chedin (4) examined the spectrum of nitrocellulose in the region of the fundamental OH frequencies. Confirming the observations of Nikitin, they found that in nitrocellulose nitrated to 6.7, 11.2, and 13.8% nitrogen, the free OH progressively increased owing to the increasing distance between the chains.

Kagarise and Weinberger (20) recorded the spectrum obtained by evaporating a methyl ethyl ketone solution of nitrocellulose. They noted a broad hydroxyl band at 2.95 microns, a weak absorption band at 3.45 microns indicating a small number of carbon-hydrogen linkages, strong bands at 6.05 and 7.82 microns characteristic of the nitrate group, and a strong band at 11.92 microns in some manner related to the nitrate group since it did not appear in other cellulosics.

Pristera, Halik, Castelli, and Fredericks (33) recorded the spectrum of a nitrocellulose film cast from cyclohexanone solution.

Sadtler Research Laboratories (40) recorded the spectra of nitrocellulose films cast from acetone solutions.

Guthrie and Spedding (14) in a study of carbohydrate nitrates and sulfonates prepared trinitrocellulose film by nitrating a very thin viscose film with a mixture of nitric acid, phosphoric acid, and phosphorus pentoxide. They noted bands at 1656 cm^{-1}, 1276 cm^{-1}, 840 cm^{-1}, 746 cm^{-1}, and 690 cm^{-1}. The first three bands were easier to identify by reason of their high intensity and singularity than were the last two. The bands at 1656 cm^{-1} and 1276 cm^{-1} corresponded to the asymmetric and stretching frequencies of the NO_2 grouping. The other bands were in some way related to the nitrate group.

Phillips, Orlick, and Steinberger (30) investigated the kinetics of the thermal decomposition of nitrocellulose by measuring the nitrate bands at 6 microns, 7.8 microns and 12 microns, and the "C-O-C" band (related to the basic cellulose skeleton) at 9.35 microns. They used films prepared by evaporating acetone solutions.

Rosenberger and Shoemaker (38) determined nitrocellulose in mixtures of cellulose resins by dissolving in acetone and measuring the absorbance at 11.92 microns. The accuracy was fair, as indicated by the fact that the mean deviation was about ± 0.4% nitrocellulose.

Kuhn (21) recorded a spectrum of nitrocellulose film cast from ethyl acetate solution and described experiments on the determination of nitrogen in nitrocellulose. He found that the band at 6 microns could not be used for the determination because even very thin films showed almost complete absorption at this wave length. He therefore chose the hydroxyl band at 3 microns for the determination (it will be recalled that the higher the nitrate content the lower the hydroxyl content). To overcome the error caused by the variation in film thickness, he computed the ratio of the hydroxyl band at 3 microns to the carbon-hydrogen band at 3.5 microns (the assumption was made that the intensity of the latter band was not changed by increasing nitration). The accuracy of the proposed method was limited. Kuhn noted the difficulty of completely removing the solvent from films.

The infrared spectra of previous investigators showed fairly good agreement in the range of 2 to 15 microns (the region to which the sodium chloride optics are applicable). The range between 1 and 2 microns seemed controversial.

To sum up the significance of the bands in the range of 2 to 15 microns, it can be stated that the large bands at 6.0 microns, 7.8 microns, and 11.9 microns are due to the nitrate group, as are the smaller bands at 13.3 and 14.3 microns. The weak band at 2.9 to 3.0 microns can be ascribed to the hydroxyl group and is due to the fact that the cellulose has not been completely nitrated. The medium band at 3.4 to 3.5 microns is due to the carbon-hydrogen linkage, and the band at 9.3 to 9.4 is probably due to the "C-O-C" grouping. The significance of the other smaller bands in the 2 to 15 micron region has apparently not been explained.

C. DEVELOPMENT OF PROPOSED METHOD FOR THE DETERMINATION OF NITROGEN IN NITROCELLULOSE BY INFRARED

It seemed to the authors that it was not feasible to use the film technique for the determination of nitrogen in nitrocellulose because of the difficulties of measuring the thickness of the film and removing all the solvent and water.

Some experiments were conducted using the potassium bromide pellet technique. The spectra obtained for different concentrations of a sample of nitrocellulose (12.3% nitrogen) in potassium bromide are shown in Figure 1.

The potassium bromide discs (0.85 mm in thickness) were made by use of an evacuable die manufactured by Perkin-Elmer Corporation; the spectra were recorded by means of a Beckman IR2T Infrared Spectrophotometer. The spectra would seem to indicate that the 0.1% concentration would be the most satisfactory concentration for determining nitrogen by the pellet technique. The results obtained for nitrogen, however, using this concentration, were not entirely satisfactory so the pellet technique was abandoned. It is to be noted that the spectrum obtained for nitrocellulose by the pellet technique resembled that obtained by the film technique by previous investigators.

The most feasible method of approach seemed to be to dissolve the nitrocellulose in a solvent and measure the absorbance at one of the three sharp bands due to the nitrate.

Many solvents have been used for dissolving nitrocellulose including the following: esters (such as ethyl acetate), acetone, methyl ethyl ketone, cyclohexanone, dioxane, methyl alcohol, nitrobenzene, nitroethane, tetra-hydrofuran (9, 10), propylene oxide, pyridine, and a mixture of ethyl alcohol and ether.

In order to be applicable to the determination of nitrogen in nitrocellulose, the solvent had to completely dissolve samples of high nitrogen content as well as samples of lower nitrogen content and show small absorption at a wave length that could be used for measuring a nitrate band.

Most solvents for nitrocellulose do not meet both of these conditions. For instance, methyl alcohol, or a mixture of ethyl alcohol and ether will not completely dissolve samples containing more than about 13% nitrogen. Nitrobenzene and nitroethane are very good solvents but are eliminated because of their composition. Many esters are satisfactory insofar as the dissolution process is concerned but they do not show a small enough absorbance at the wave lengths of the nitrate bands. This is illustrated in Figure 2 by the spectrum of ethyl acetate obtained using a 0.2 mm cell (previous investigators have reported the spectrum using smaller width cells). Some consideration was given to the use of acetone as the solvent at 11.9 microns. However, the results obtained were only fair. The transmittance of acetone at 11.9 microns using an 0.2 mm cell was about 74% (Figure 3). The spectrum of acetone indicated that acetone could not be used at 6 microns or 7.8 microns.

The most satisfactory solvent was tetrahydrofuran (also known as tetrahydrofurane, tetramethylene oxide, and tetramethylene ether). Since this solvent is somewhat unusual, its properties were considered in some detail. It is a cylic ether, $(CH_2CH_2)_2O$, made by the catalytic hydrogenation

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of furan. The cost of the high purity material (obtainable from Eastman Kodak Co. and Fisher Scientific Co.) is roughly twice that of reagent grade acetone. The boiling point is 66° C. and the specific gravity is 0.888. A study of its vapor pressure has been made by DuPont (10). The water content of the high purity solvent is about 0.02% (11) (the water content of reagent acetone is about 0.5%) and the solvent is miscible with water in all proportions. Tetrahydrofuran is shipped in brown bottles and contains an antioxidant to prevent the formation of peroxides (Eastman Kodak Co. uses a 0.1% of hydroquinone). The danger from peroxide explosions is much less with tetrahydrofuran than with some other ethers (for instance, isopropyl ether) (15, 34). Latest researches would seem to indicate that tetra-hydrofuran is probably slightly more toxic than ethyl ether and definitely less toxic than dioxane (18).

The infrared spectrum of tetrahydrofuran using 0.03 mm cell has been reported (3, 41). The spectrum obtained in this laboratory, using a 0.2 mm cell is shown in Figure 4. The absorption of tetrahydrofuran at 6 microns was about 87% so the use of the nitrate band at this wave length seemed feasible.

It was not necessary to distill the tetrahydrofuran or dry it. Distilled tetrahydrofuran gave a spectrogram that did not differ significantly from that obtained for the undistilled solvent. Tetrahydrofuran dried for several days by treatment with anhydrous sodium sulfate and sodium chloride (32) gave a spectrogram that did not differ appreciably from that of the untreated solvent. Greinacher, Lüttke, and Mecke (13) made a study of the effect of water on the spectrum of tetrahydrofuran (and other solvents) and found that water in tetrahydrofuran produced bands at about 2.8 and 6 microns.

The development of the actual working method was not as simple as originally thought. It was found that satisfactory results could only be obtained by allowing the samples to dissolve by standing overnight. At first, the samples were dissolved by stirring with a magnetic stirrer for 30 minutes, but the data obtained left much to be desired. The failure of the stirring technique may be explained by the fact that the dissolution of nitrocellulose is a peculiar process that does not proceed as with low molecular weight organic compounds, but is preceded by swelling during which the volume of the nitrocellulose increases considerably (1). This swelling is a slow process that is not readily hastened by agitation or heat (1). Other reasons for the failure of the rapid stirring process could be that agitation causes depolymerization of the nitrocellulose (1, 22) and the fact that the properties of nitrocellulose solutions change with time (1, 22).

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An exhaustive investigation of the stirring technique could possibly establish conditions for the successful use of that procedure. Such a study would require a battery of magnetic stirrers which was not available to this laboratory.

To obtain maximum accuracy and reproducibility, it was necessary to control the concentration of the solution at approximately 0.78 gram of nitrocellulose p̄ 100 grams of solution. This was easily done by weighing out 0.30 to 0.32 gram of the nitrocellulose into a glass-stoppered Erlenmeyer flask and adding 45 ml of tetrahydrofuran measured with a tall-type 50 ml graduate. The weights of tetrahydrofuran (weights of solutions minus weights of nitrocellulose) obtained on a series of samples were 39.325, 39.414, 39.438, 39.227, and 39.565 grams. These figures showed that this method of measuring the tetrahydrofuran was satisfactory. Tetrahydrofuran is fairly volatile so the usual precautions to prevent excessive volatilization should be taken (for instance, the solutions should not be kept under a hood). The error caused by volatilization is minimized inasmuch as the weight of the solution is recorded to five significant figures whereas the percentage of nitrogen is reported to only four significant figures.

There are two reasons why the concentration should be controlled as suggested. First, the best accuracy and maximum change in absorbance per mg of nitrogen was obtained at this concentration (using a 0.2 mm cell) for the range of nitrogen in which this laboratory was interested (12 to 13.8%). The recommended concentration gave a transmittance of nitrocellulose plus solvent of about 18 to 23%. It has been pointed out by statistical treatment that a range of 20 to 6 0% in the transmittance will give almost equivalent accuracy for the usual infrared analysis (36, 37).

Secondly, the concentration of the solution is such that there is considerable solvation when nitrocellulose is dissolved (i.e., the nitrocellulose molecules are surrounded by layers of solvent molecules) (1). Apparently, the concentration affects the degree of solvation somewhat and this in turn affects the absorbance.

It was necessary to control the temperature of the tetrahydrofuran solutions in order to obtain maximum accuracy. This was done by allowing the dissolution process to take place in the air conditioned room which housed the infrared instrument. An alternative method might be to use a constant temperature bath. The errors of temperature cannot be eliminated by placing the solution in the cell and allowing the cell to stand for several minutes since this introduces a new factor, namely the change in volume of the solution in the cell.

In laboratories not possessing an air-conditioned room for the infrared instrument or a constant temperature bath, a new curve could be drawn up for every batch of samples. Since the working time per sample is very small, this would not be too objectionable.

The mode of calculation was relatively simple. The spectrum of each sample was run from 5.80 to 6.05 microns and the transmittance of top of the left hand arm of the band (I_A) and the transmittance of the bottom of the band (I) (see A, Figure 5) were noted. The ratio of I_A to I was taken and log (I_A/I) was related to the nitrogen concentration (mg of nitrogen per gram of solution). In effect this method of calculation amounts to subtracting the absorbance of the top of the band (A_A) from the absorbance at the bottom of the band (A), since log (I_A/I)=A-A_A. The use of transmittance scale is preferable to the absorbance scale because the former scale can be read more accurately than the absorbance scale in the region of 20% transmittance.

Other modes of calculation and other techniques were considered. The base line technique (6, 42) whereby the spectrum was run from 5.80 to 6.25 microns (B, Figure 5) and log (I_B/I) related to the concentration seemed subject to more errors of measurement for this determination. Another method for making the calculation would be actually to deduct for the solvent blank. The curve for the cell plus solvent over the range of 5.80 to 6.25 microns is shown in C, Figure 5 (of course, the whole curve for the blank need not be run but only the point at which the nitrate peak is measured). Still another technique would be to place a matched cell containing tetrahydrofuran in the reference beam. The curve obtained when a cell of approximately the same dimensions as the sample cell was placed in the reference beam and the spectrum run from 5.60 to 6.45 microns is shown in D, Figure 5. Possibly, the matched cell method might be slightly more accurate than the suggested method, but inasmuch as matched cells were not available throughout the course of this work, this technique was not considered. A disadvantage in the use of the matched cell technique is the fact that cells change with use.

The exact peak of the nitrate band in the tetrahydrofuran solution spectrum occurred at about 6.00 microns (in the film spectrum it occurred at about 6.05 microns and in the potassium bromide pellet spectrum at about 6.02 microns). The peak of this band was much sharper in the tetrahydrofuran solution spectrum than in the film spectrum or potassium bromide pellet spectrum. This was to be expected.

A straight line calibration curve was obtained on carrying standard samples of nitrocellulose through the recommended procedure. As stated previously, the range of this curve was 12 to 13.8% nitrogen. For 10.5 to 12% nitrogen, a different calibration curve might have to be prepared. This was not investigated.

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D. RECOMMENDED METHOD

1. Preparation of Calibration Curve

Place about 2 grams of nitrocellulose standards (preferably 3 or more) in weighing bottles and dry at 65° C. at 2 to 5 cm pressure for 4 hours. Cover and allow to cool in a desiccator containing a good desiccant for 30 minutes or more.

Rinse 125 ml Erlenmeyer flasks with glass stoppers (No. 22) with acetone and shake out the excess acetone. After treating the flasks with solvent always place them on a clean surface not attacked by the solvent (for instance, paper towels). Dry the flasks and stoppers by heating in an oven at 130° C. for 1 hour or more. Cover with the stoppers, allow to stand in the atmosphere for 30 minutes or more, and weigh to the nearest mg. The same weights can be used for all subsequent determinations. Care should be taken not to interchange the stoppers.

Place 0.31 to 0.32 gram of the sample in an aluminum scoop-type balance pan and weigh to the nearest 0.1 mg. Pour the sample into the Erlenmeyer flask and tap the scoop. Tap the flask against the table top, brush down any particules that adhere in the mouth of the flask, tap the flask again, and insert the stopper. Weigh the balance pan plus residual nitrocellulose and calculate the exact weight of the sample.

Pour 45 ml of high purity tetrahydrofuran, measured with a 50 ml tall-type graduate, into the flasks. Do not pour the solvent around the sides of the flasks since this will cause volatilization losses. Cover with the stoppers and allow to stand overnight at the temperature of the room containing the infrared instrument.

Swirl the flasks and weigh to the nearest mg. Calculate the weights of the solutions.

Rinse a 0.2 mm infrared cell once with tetrahydrofuran and three times with the solution of the sample, using a 1 cc syringe pipet. Fill the cell with a solution of the sample and run the infrared spectrum from 5.80 to 6.05 microns. For the Perkin-Elmer Model 21, use the following settings: Resolution 941, Speed 1, Gain 5, Response 2, Suppression 0, approximately 0.5 micron per minute (the 5.80 and 6.05 micron readings for the wave length corresponded to slit widths of 0.143 and 0.149 mm respectively).

Observe I_A and I (see A, Figure 5) and calculate log (I_A/I).

Calculate the nitrogen concentrations as follows:

$$\text{Mg of N per gram of solution} = \frac{\text{grams of NC x \% N in NC x 10}}{\text{grams of solution}}$$

Plot log (I_A/I) against mg of nitrogen per gram of solution, using standard graph paper.

Pour the contents of the flasks into a liter beaker containing tap water, wash the flasks and stoppers thoroughly with acetone, and dry in an oven at 130° C. for 1 hour or more.

2. Procedure

Place 0.31 to 0.32 gram of the sample into the balance pan and proceed as described under Preparation of Calibration Curve to the calculation of log (I_A/I). Convert the reading of log (I_A/I) to mg of nitrogen per gram of solution by consulting the calibration curve.

Calculate the percent nitrogen as follows:

$$\text{\% N in NC} = \frac{\text{mg of N per gram of solution x grams of solution}}{\text{grams of NC x 10}}$$

E. RESULTS

The results obtained by the proposed method for five samples of nitro-cellulose that had been accurately analyzed by the nitrometer method by Picatinny Arsenal are shown in Table I. The results for the proposed method compare well with the results by the nitrometer method. The precision for the proposed method (average about ± 0.09% is satisfactory but is not as good as that obtainable by the nitrometer method in the hands of an experienced operator. The source of the nitrocellulose did not affect the results obtained by the infrared method.

F. ACKNOWLEDGMENTS

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Table I. Results for Nitrogen in Nitrocellulose

Sample	Source	Nitrogen Content by Nitrometer (%)		Nitrogen Content by Infrared (%)
1	Wood pulp	12.28		12.28
				12.17
				12.24
				12.29
				12.31
			Avg.	12.26
			Std. Dev.[a]	0.055
2	Wood pulp	13.19		13.12
				13.23
				13.29
				13.08
				13.09
			Avg.	13.16
			Std. Dev.	0.093
3	Cotton linters	13.27		13.32
				13.27
				13.15
				13.06
				13.28
			Avg.	13.22
			Std. Dev.	0.108
4	Cotton linters	12.24		12.15
				12.29
				12.12
				12.30
				12.27
			Avg.	12.23
			Std. Dev.	0.085
5	Cotton linters	12.64		12.63
				12.91
				12.70
				12.62
				12.61
			Avg.	12.69
			Std. Dev.	0.126

[a]
$$\sqrt{\frac{d_1^2 + d_2^2 + \ldots \, d_n^2}{n-1}}$$

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16 - Commanding Officer
     Frankford Arsenal
     Philadelphia 37, Pa.
     ATTN:  Thru;  Chief, 1000
                   Dep. Chief, 1000                    (1)
            Thru;  Chief, 1100
                   Dep. Chief for Res., 1100           (1)
            Chief, 1700                                 (1)
            Thru;  Director of Res. & Dev., 0100
                   Commanding Officer                   (1)
            1700, L. H. Adam                            (1)
            J. McGrath, 6120                            (1)
            M. A. Fry, 5500                             (1)
            L. Charno, 6152                             (1)
            A. Frank, 6152                              (1)
            Chief, 1720                                 (1)
            Chief, 1721                                 (2)
            Chief, 1723                                 (1)
            H. Levitsky                                 (1)
            J. Cohen, 1723                              (2)
            G. Norwitz, 1723                            (2)
```

1 - Atlantic Research Corporation
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    Alexandria, Virginia
    ATTN: Mr. B. W. Black

1 - E. I. du Pont de Nemours and Company
    Carneys Point Process Laboratory
    P. O. Box 152
    Penns Grove, New Jersey
    ATTN: Mr. C. I. Johnson

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    Air Force Flight Test Center
    Edwards Air Force Base, Calif.
    ATTN: FTRSC
           Lt. H. V. Bankaitis

1 - Hercules Powder Company
    Allegany Ballistics Laboratory
    P. O. Box 210
    Cumberland, Maryland
    ATTN: Mr. W. E. Kight

1 - Hercules Powder Company
    Kenvil, New Jersey
    ATTN: Mr. H. A. Read

1 - National Research Corporation
    70 Memorial Drive
    Cambridge 42, Massachusetts
    ATTN: Dr. J. H. Atkins

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    White Oak, Silver Spring, Md.
    ATTN: Dr. J. M. Rosen
           Organic Chemistry Div.

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    China Lake, California
    ATTN: Mr. R. H. Pierson, Code 5052
           Mr. E. Julien, Code 4521

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   U. S. Naval Propellant Plant
   Indian Head, Maryland
   ATTN: Mr. H. L. Stalcup
          Dr. Mae Fauth

1 - Headquarters
   Ogden Air Material Area
   Hill Air Force Base, Utah
   ATTN: Mr. Neal M. Hansen

1 - Olin Mathieson Chemical Corporation
   P. O. Box 508
   Marion, Illinois
   ATTN: Mr. R. J. Thiede

1 - Olin Mathieson Chemical Corporation
   Explosives Division
   Quality Control Department
   East Alton, Illinois

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   Picatinny Arsenal
   Dover, New Jersey
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          Feltman Res. and Eng. Lab.
          ORDBB-TM7-001
          Mr. A. Sokol, ORDBB-DC7

1 - Commanding Officer
   Ordnance Corps
   Radford Arsenal
   Radford, Virginia
   ATTN: Mr. E. G. Simpson
          (Hercules Powder Company)

1 - Commander, OML
   Army Rocket and Guided Missile Agency
   Redstone Arsenal, Alabama
   ATTN: Mr. W. W. Howard, Bldg. #7120

1 - Sandia Corporation
   P. O. Box 5800
   Albuquerque, New Mexico
   ATTN: Mr. R. J. Buxton, Code 1625

1 - Dr. R. F. Muraca
   Stanford Research Institute
   Poulter Laboratories
   Menlo Park, California

1 - Dr. Eugene Burns
   Stanford Research Institute
   Poulter Laboratories
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          Mr. Charles G. Hain

1 - Canadian Armament Research and
   Development Establishment
   P. O. Box 1427
   Quebec, P. Q., Canada
   ATTN: Mr. W. L. Phene

1 - Canadian Arsenals, Ltd.
   Valleyfield, Quebec, Canada
   ATTN: Mr. C. R. Lee

2 - Inspection Services of Canada
   75 Patrick Street
   Ottawa, Ontario, Canada
   ATTN: Mr. H. A. Showalter

1 - Ministry of Aviation
   Chemical Inspectorate
   Headquarters Building
   Royal Arsenal
   Woolwich, London, S. E. 18
   ATTN: Mr. J. J. Lamond

1 - Ministry of Supply
   Chemical Inspectorate
   Bishopton, Renfrewshire,
   Scotland

3 - Chief of Ordnance
    Department of the Army
    Washington 25, D. C.
    ATTN: ORDIM, Eng. Section
          ORDTX-AR
          ORDTS

1 - Commanding General
    Aberdeen Proving Ground
    Aberdeen, Maryland
    ATTN: DRL, ORDBG-BLI

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    Inyokern, California
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    Washington 25, D. C.
    ATTN: Section Re2a

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    Ordnance Training Ground
    Aberdeen Proving Ground, Maryland

1 - U.S. Atomic Energy Commission
    Division of Military Application
    Washington 25, D. C.

10 - Commander
     Armed Services Technical Info Agy
     Arlington Hall Station
     Arlington 12 Virginia
     ATTN: TIPDR

2 - Solid Propellant Information Agy
    Applied Physics Laboratory
    The Johns Hopkins University
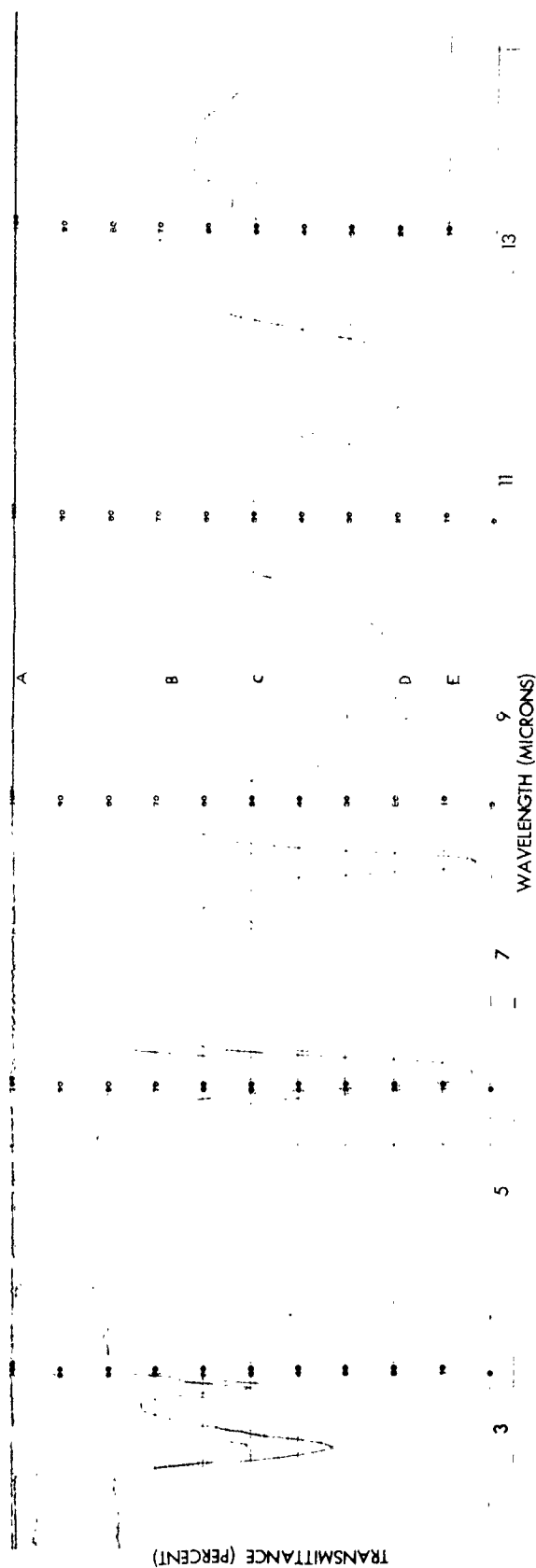   8621 Georgia Avenue
    Silver Spring, Maryland

Figure 1. Infrared spectra of nitrocellulose using the potassium bromide pellet technique

A. Blank
B. 0.05% nitrocellulose in potassium bromide
C. 0.10% nitrocellulose in potassium bromide
D. 0.20% nitrocellulose in potassium bromide
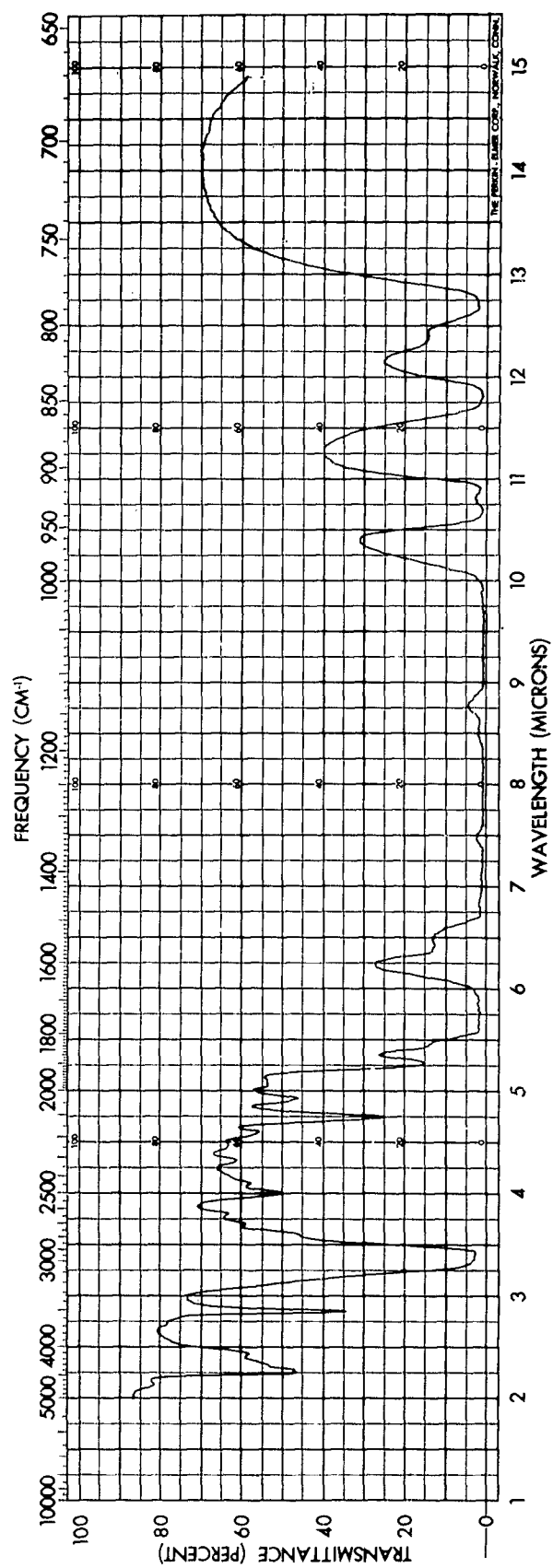E. 0.50% nitrocellulose in potassium bromide
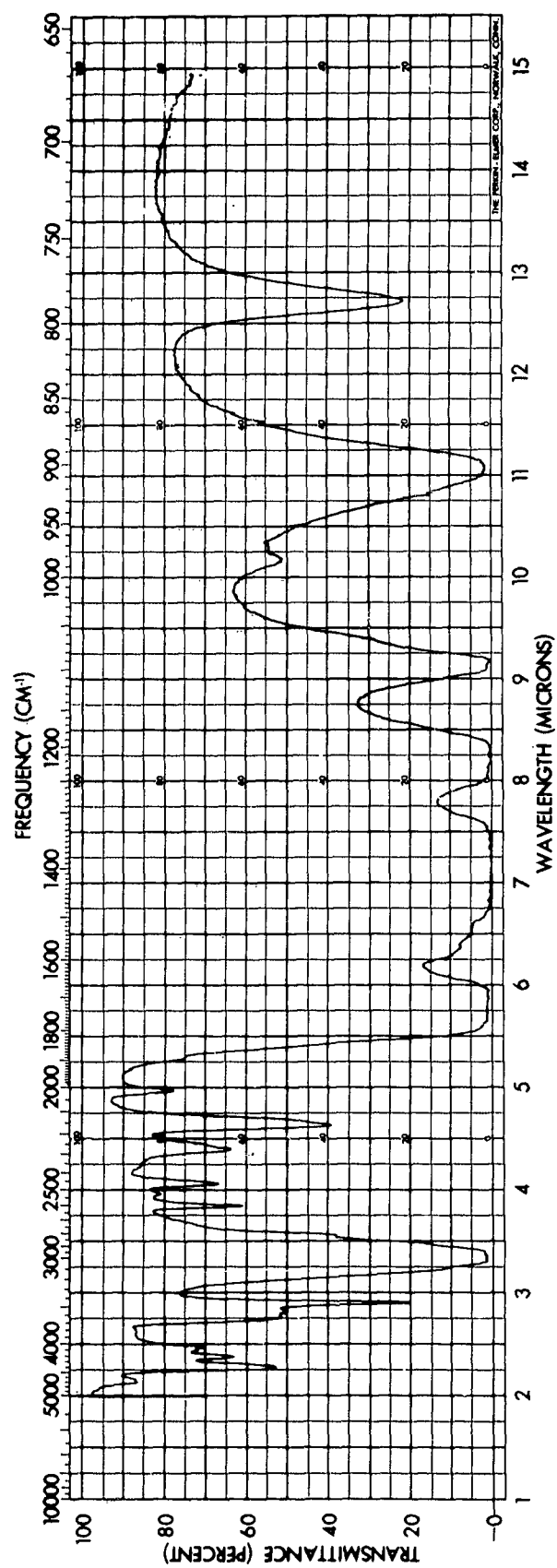
Figure 2. Infrared spectrum of ethyl acetate (0.2 mm cell)

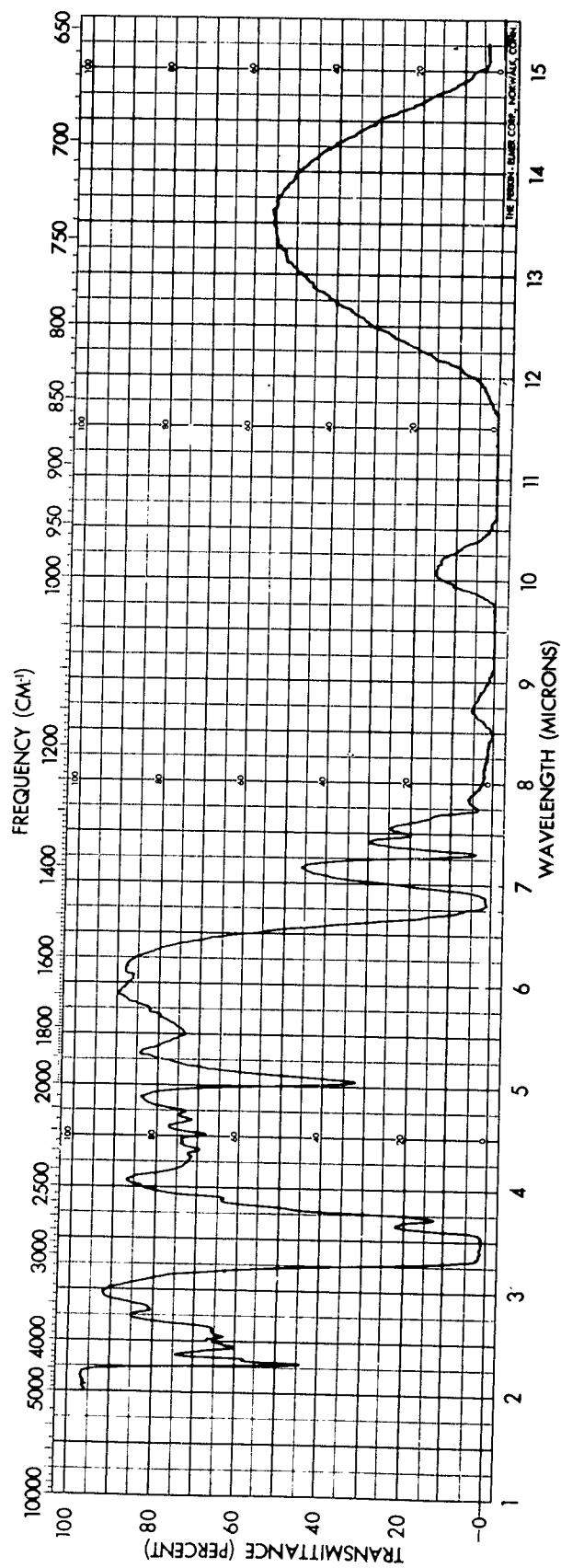Figure 3.   Infrared spectrum of acetone (0.2 mm cell)

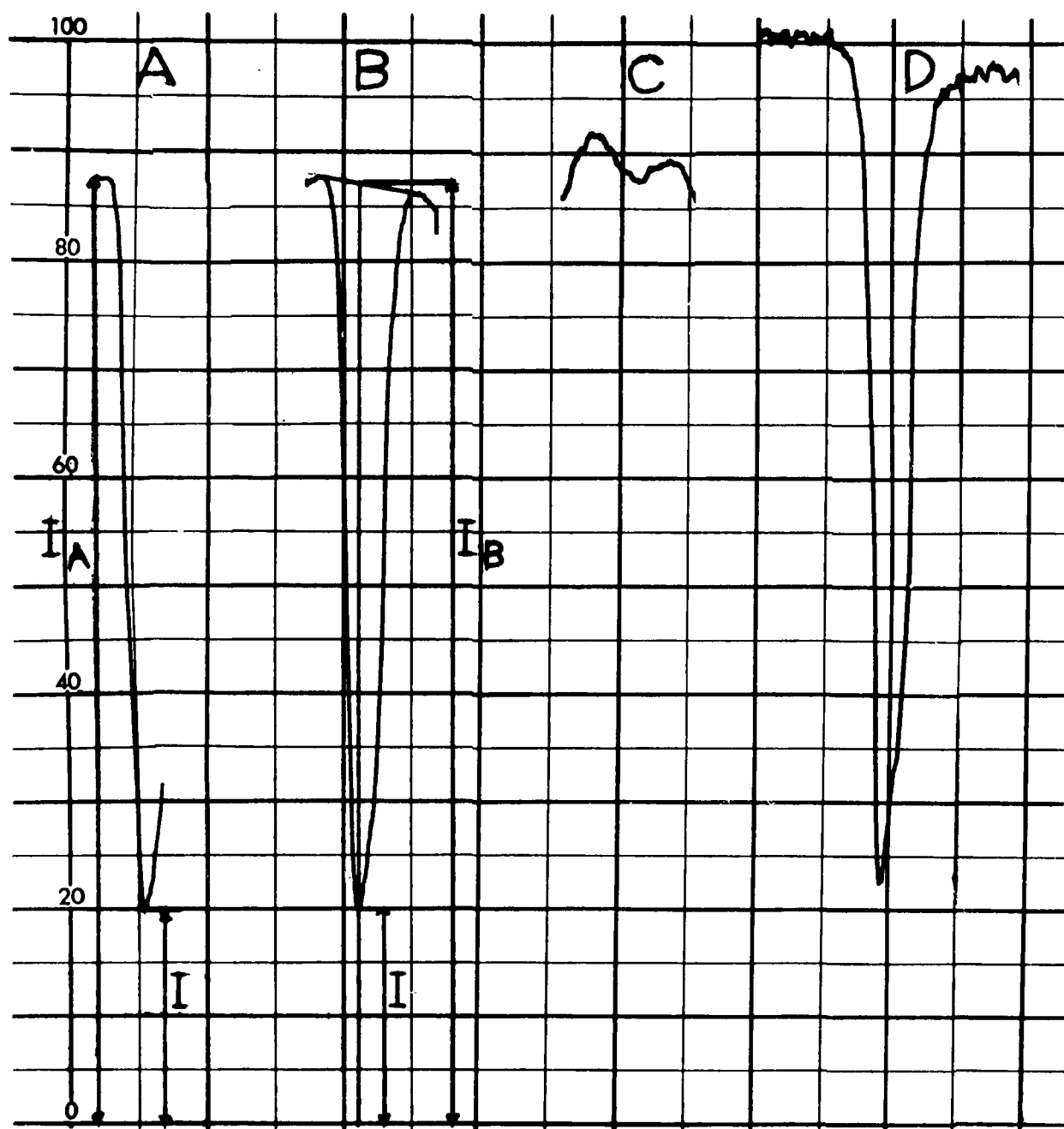Figure 4. Infrared spectrum of tetrahydrofuran (0.2 mm cell)

Figure 5.   Infrared spectra at approximately 6 microns

A.   Spectrum of nitrocellulose in tetrahydrofuran from 5.80 to 6.05 microns
B.   Spectrum of nitrocellulose in tetrahydrofuran from 5.80 to 6.25 microns
C.   Spectrum of blank (tetrahydrofuran) from 5.80 to 6.25 microns
D.   Spectrum of nitrocellulose in tetrahydrofuran from 5.60 to 6.45 microns
       with tetrahydrofuran in reference beam